

518 Rec'd PCT/PTO 23 JUL 2001

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| U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE | | ATTORNEY'S DOCKET NUMBER ANGELI=2 |
| TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371 | | U.S. APPLICATION NO (If known, see 37 CFR 1.5) 09/889830 |
| INTERNATIONAL APPLICATION NO. PCT/FR00/00118 | INTERNATIONAL FILING DATE 20 January 2000 | PRIORITY CLAIMED 21 January 1999 |
| TITLE OF INVENTION CONCENTRATED OR DILUTABLE SOLUTIONS OR DISPERSIONS, PREPARATION... | | |
| APPLICANT(S) FOR DO/EO/US Jean-Marc ANGELI et al. | | |
| <p>Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:</p> <ol style="list-style-type: none">1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).4. <input checked="" type="checkbox"/> The US has been elected in a Demand by the expiration of 19 months from the priority date (PCT Article 31).5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))<ol style="list-style-type: none">a. <input type="checkbox"/> is attached hereto (required only if not transmitted by the International Bureau).b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))<ol style="list-style-type: none">a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).b. <input type="checkbox"/> have been communicated by the International Bureau.c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.d. <input checked="" type="checkbox"/> have not been made and will not be made8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). <p>Items 11. to 16. below concern document(s) or information included:</p> <ol style="list-style-type: none">11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.12. <input type="checkbox"/> An Assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.14. <input type="checkbox"/> A substitute specification.15. <input type="checkbox"/> A change of power of attorney and/or address letter.16. <input checked="" type="checkbox"/> Other items or information:<ul style="list-style-type: none"><input checked="" type="checkbox"/> Courtesy copy of the first page of the International Publication (WO 00/43117).<input checked="" type="checkbox"/> Courtesy copy of the International Preliminary Examination Report (In French). There were no annexes.<input checked="" type="checkbox"/> Courtesy Copy of the International Search Report.<input checked="" type="checkbox"/> Application Data Sheet <p><input checked="" type="checkbox"/> The application is (or will be) assigned to: <u>ARCANE INDUSTRIES</u>, whose address is <u>Z.I. des Paluds, 73, avenue du Douard, F-13685 Aubagne, France.</u></p> | | |

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| U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/889830 | International Application No. PCT/FR00/00118 | Attorney's Docket No. ANGELI=2 |
|---|--|--|

17. [xx] The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a)(1)-(5):

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO.....\$1000.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO... \$860.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....\$710.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4).....\$690.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4).....\$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

Surcharge of \$130.00 for furnishing the oath or declaration later than [] 20 [X] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).

| Claims as Originally Presented | Number Filed | Number Extra | Rate |
|---|--------------|--------------|-----------|
| Total Claims | 10 - 20 | | X \$18.00 |
| Independent Claims | 1 - 3 | | X \$80.00 |
| Multiple Dependent Claims (if applicable) | | | +\$270.00 |

TOTAL OF ABOVE CALCULATIONS =

| Claims After Post Filing Prel. Amend | Number Filed | Number Extra | Rate |
|--------------------------------------|--------------|--------------|-----------|
| Total Claims | - 20 | | X \$18.00 |
| Independent Claims | - 3 | | X \$78.00 |

TOTAL OF ABOVE CALCULATIONS =

Reduction of 1/2 for filing by small entity, if applicable. Applicant claims small entity status. See 37 CFR 1.27.

SUBTOTAL =

Processing fee of \$130.00 for furnishing the English translation later than [] 20 [] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).

TOTAL NATIONAL FEE =

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

TOTAL FEES ENCLOSED =

| | |
|---------------|----|
| Amount to be: | \$ |
| refunded | |
| charged | \$ |

- a. [] A check in the amount of \$_____ to cover the above fees is enclosed.
- b. [X] Credit Card Payment Form (PTO-2038), authorizing payment in the amount of \$ 990.00, is attached.
- c. [] Please charge my Deposit Account No. 02-4035 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.
- d. [XX] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4035. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

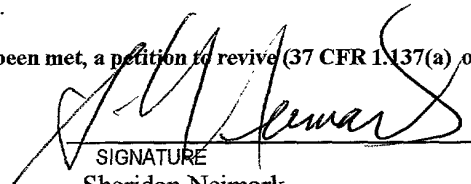
BROWDY AND NEIMARK, P.L.L.C.
624 NINTH STREET, N.W., SUITE 300
WASHINGTON, D.C. 20001

TEL: (202) 628-5197

FAX: (202) 737-3528

Date of this submission: **MONDAY, July 23, 2001**

Form PTO-1390 (as slightly revised by Browdy and Neimark)


SIGNATURE
Sheridan Neimark
NAME
20,520
REGISTRATION NUMBER

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

ATTY.'S DOCKET: ANGELI=2

| | | |
|-----------------------------|---|------------------|
| In re Application of: |) | Art Unit: |
| Jean-Marc ANGELI et al. |) | |
| |) | Examiner: |
| |) | |
| I.A. No.: PCT/FR00/00118 |) | Washington, D.C. |
| |) | |
| I.A. Filed: 20 January 2000 |) | July 23, 2001 |
| |) | |
| For: CONCENTRATED OR.. |) | |

PRELIMINARY AMENDMENT

Honorable Commissioner for Patents and Trademarks
Washington, D.C. 20231

Sir:

Contemporaneous with the filing of this case and
prior to calculation of the filing fee, kindly amend as
follows:

IN THE SPECIFICATION

After the title please insert the following
paragraph:

REFERENCE TO RELATED APPLICATIONS

--The present application is the national stage
under 35 U.S.C. §371 of international application
PCT/FR00/00118, filed January 20, 2000 which designated the
United States, and which application was not published in the
English language.--

204150 02000000

IN THE CLAIMS

2. (Amended) A concentrated composition as defined in claim 1, characterised in that the hydrophobic active agent is a hydrophobic active agent with a KB index greater than or equal to 40.

3. (Amended) A concentrated composition as defined in claim 1, characterised in that it contains a mixture of two glycol ethers.

4. (Amended) A concentrated composition as defined in claim 1, characterised in that it is in the form of a micro-emulsion.

5. (Amended) A concentrated composition as defined in claim 1, characterised in that it contains dipropylene glycol methyl ether as a glycol ether.

7. (Amended) A composition as defined in claim 1, characterised in that it contains 50 % to 90 % by weight of glycol ether or ethers.

8. (Amended) A composition as defined in claim 1, characterised in that it is diluted so that the diluent phase represents up to 99 % by weight of the micro-emulsion or diluted solution.

9. (Amended) A method of preparing a concentrated composition as defined in claim 1, characterised in that the liquid active agent or active agents are mixed under agitation

In re of: Jean-Marc ANGELI et al. (ANGELI=2)

with the glycol ether or ethers in order to emulsify or solubilize the latter.

10. (Amended) A washing or stripping agent comprising a concentrated or diluted composition as defined in claim 1.

REMARKS

The above amendment to the specification is being made to insert reference to the PCT application of which the present case is a U.S. national stage. The above amendments to the claims are being made in order to eliminate any properly multiply dependent claims, for the purpose of reducing the filing fee. Please enter this amendment prior to calculation of the filing fee in this case.

Attached hereto is a marked-up version of the changes made to the specification and claims by the current amendment. The attached page is captioned "Version with Markings to Show Changes Made."

Favorable consideration is earnestly solicited.

Respectfully submitted,
BROWDY AND NEIMARK, P.L.L.C.
Attorneys for Applicant

By: 

Sheridan Neimark

Registration No. 20,520

SN:wrđ

Telephone No.: (202) 628-5197

Facsimile No.: (202) 737-3528

VERSION WITH MARKINGS TO SHOW CHANGES MADE

2. A concentrated composition as defined in claim 1, characterised in that the hydrophobic active agent is a hydrophobic active agent with a KB index greater than or equal to 40.

3. A concentrated composition as defined in claim ~~1 or 2~~, characterised in that it contains a mixture of two glycol ethers.

4. A concentrated composition as defined in ~~one of claims 1 to 3~~claim 1, characterised in that it is in the form of a micro-emulsion.

5. A concentrated composition as defined in ~~one of claims 1 to 4~~claim 1, characterised in that it contains dipropylene glycol methyl ether as a glycol ether.

7. A composition as defined in ~~one of claims 1 to 6~~claim 1, characterised in that it contains 50 % to 90 % by weight of glycol ether or ethers.

8. A composition as defined in ~~one of claims 1 to 6~~claim 1, characterised in that it is diluted so that the diluent phase represents up to 99 % by weight of the micro-emulsion or diluted solution.

9. A method of preparing a concentrated composition as defined in ~~one of claims 1 to 7~~claim 1, characterised in that the liquid active agent or active agents are mixed under agitation with the glycol ether or ethers in order to emulsify or solubilize the latter.

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10. A washing or stripping agent comprising a concentrated or diluted composition as defined in ~~one of~~ claims 1 to 8claim 1.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

ATTY.'S DOCKET: ANGELI=2

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| In re Application of: |) | Art Unit: |
| Jean-Marc ANGELI et al. |) | |
| |) | Examiner: |
| |) | |
| I.A. No.: PCT/FR00/00118 |) | Washington, D.C. |
| |) | |
| I.A. Filed: 20 January 2000 |) | July 23, 2001 |
| |) | |
| For: CONCENTRATED OR... |) | |

PRELIMINARY AMENDMENT

Honorable Commissioner for Patents and Trademarks
Washington, D.C. 20231

Sir:

Contemporaneous with the filing of this case and
prior to calculation of the filing fee, kindly amend as
follows:

IN THE SPECIFICATION

After the title please insert the following
paragraph:

REFERENCE TO RELATED APPLICATIONS

--The present application is the national stage
under 35 U.S.C. §371 of international application
PCT/FR00/00118, filed January 20, 2000 which designated the
United States, and which application was not published in the
English language.--

204450-0286860

IN THE CLAIMS

3. (Amended) A concentrated composition as defined in claim 1, characterised in that it contains a mixture of two glycol ethers.

4. (Amended) A concentrated composition as defined in claim 1, characterised in that it is in the form of a micro-emulsion.

5. (Amended) A concentrated composition as defined in claim 1, characterised in that it contains dipropylene glycol methyl ether as a glycol ether.

7. (Amended) A composition as defined in claim 1, characterised in that it contains 50 % to 90 % by weight of glycol ether or ethers.

8. (Amended) A composition as defined in claim 1, characterised in that it is diluted so that the diluent phase represents up to 99 % by weight of the micro-emulsion or diluted solution.

9. (Amended) A method of preparing a concentrated composition as defined in claim 1, characterised in that the liquid active agent or active agents are mixed under agitation with the glycol ether or ethers in order to emulsify or solubilize the latter.

10. (Amended) A washing or stripping agent comprising a concentrated or diluted composition as defined in claim 1.

In re of: Jean-Marc ANGELI et al. (ANGELI=2)

REMARKS

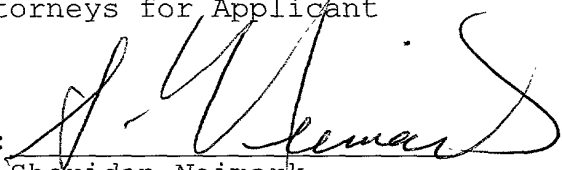
The above amendment to the specification is being made to insert reference to the PCT application of which the present case is a U.S. national stage. The above amendments to the claims are being made in order to eliminate any properly multiply dependent claims, for the purpose of reducing the filing fee. Please enter this amendment prior to calculation of the filing fee in this case.

Attached hereto is a marked-up version of the changes made to the specification and claims by the current amendment. The attached page is captioned "Version with Markings to Show Changes Made."

Favorable consideration is earnestly solicited.

Respectfully submitted,
BROWDY AND NEIMARK, P.L.L.C.
Attorneys for Applicant

By:


Sheridan Neimark

Registration No. 20,520

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Facsimile No.: (202) 737-3528

VERSION WITH MARKINGS TO SHOW CHANGES MADE

3. A concentrated composition as defined in claim ~~1 or 2~~, characterised in that it contains a mixture of two glycol ethers.

4. A concentrated composition as defined in ~~one of claims 1 to 3~~claim 1, characterised in that it is in the form of a micro-emulsion.

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8. A composition as defined in ~~one of claims 1 to 6~~claim 1, characterised in that it is diluted so that the diluent phase represents up to 99 % by weight of the micro-emulsion or diluted solution.

9. A method of preparing a concentrated composition as defined in ~~one of claims 1 to 7~~claim 1, characterised in that the liquid active agent or active agents are mixed under agitation with the glycol ether or ethers in order to emulsify or solubilize the latter.

10. A washing or stripping agent comprising a concentrated or diluted composition as defined in ~~one of claims 1 to 8~~claim 1.

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PCT

ORGANISATION MONDIALE DE LA PROPRIÉTÉ INTELLECTUELLE
Bureau international



DEMANDE INTERNATIONALE PUBLIÉE EN VERTU DU TRAITÉ DE COOPÉRATION EN MATIÈRE DE BREVETS (PCT)

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| (51) Classification internationale des brevets ⁷ : B01F 17/00 | | A1 | (11) Numéro de publication internationale: WO 00/43117 (43) Date de publication internationale: 27 juillet 2000 (27.07.00) |
| (21) Numéro de la demande internationale: PCT/FR00/00118 (22) Date de dépôt international: 20 janvier 2000 (20.01.00) (30) Données relatives à la priorité: 99/00793 21 janvier 1999 (21.01.99) FR (71) Déposant (pour tous les Etats désignés sauf US): ARCANÉ INDUSTRIES [FR/FR]; Z.I. des Paluds, 73, avenue du Douard, F-13685 Aubagne (FR). (72) Inventeurs; et (75) Inventeurs/Déposants (US seulement): ANGELI, Jean-Marc [FR/FR]; 21, Boulevard Gaston Crémieux, Résidence Périer 1er, F-13008 Marseille (FR). TESTA, Alfred [FR/FR]; Chemin de la Bastide Neuve, F-13710 Fuveau (FR). (74) Mandataire: RINUY, SANTARELLI; 14, avenue de la Grande Armée, F-75017 Paris (FR). | | | (81) Etats désignés: JP, US, brevet européen (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Publiée Avec rapport de recherche internationale. |
| (54) Title: CONCENTRATED OR DILUTABLE SOLUTIONS OR DISPERSIONS, PREPARATION METHOD AND USES (54) Titre: SOLUTIONS OU DISPERSIONS CONCENTRÉES ET DILUABLES, PROCÉDE DE PRÉPARATION ET APPLICATIONS (57) Abstract The invention concerns a composition in the form of a micro-emulsion or solution of a concentrated and dilutable active agent, characterised in that it is as clear as water and essentially consists of (a) at least 10 % of a hydrophobic active agent with a KB (Kauri butanol) index not less than 30 or an N-alkyl pyrrolidone; and (b) a glycol ether or a mixture of two glycol ethers, and in the latter case the first glycol ether having a HLB higher by 0.8 than the second glycol ether. (57) Abrégé Une composition sous forme de micro-émulsion ou solution d'un agent actif, concentrée et diluable, caractérisée en ce qu'elle est limpide comme de l'eau et qu'elle est constituée essentiellement (a) d'au moins 10 % d'un agent actif hydrophobe présentant un indice KB (Kauri butanol) supérieur ou égal à 30 ou d'une N-alkyl pyrrolidone et (b) d'un éther de glycol ou d'un mélange de deux éthers de glycol, dans ce dernier cas le premier éther de glycol présentant une HLB supérieure de 0,8 à celle du second éther de glycol. | | | |

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Concentrated and dilutable solutions or dispersions, method of preparing same and uses thereof

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The present invention relates to new concentrated and dilutable solutions or dispersions, their preparation process and to their uses.

A large number of liquid active agents can be used to effect in various fields but cannot be used as they are. In practice, because they will
10 have too strong an effect if used neat, they have to be diluted, in the form of emulsions for an active liquid agent, for example. However, this then gives rise to a problem when it comes to recovery and reprocessing, particularly if they are harmful to the environment.

Conventionally, the dispersion of an organic solvent in an
15 aqueous continuous phase in order to produce an emulsion requires the use of surfactants which are emulsifying in nature, hydrotropic agents such as xylene, toluene, or cumene sulphonate of sodium, and, more often than not, a third polar solvent such as isopropanol or glycol ethers. By way of illustration, a standard micro-emulsion of terpenic solvent will typically have the following
20 composition: water / polyoxyethylenated fatty alcohols / polyoxyethylenated nonylphenols / sodium xylene sulphonate / isopropanol / terpenic solvent. Its life is limited, as is its temperature resistance. It can thus only be used cold.

Conventionally, micro-emulsions are obtained by progressively adding an aqueous phase to an organic phase (or vice versa) under constant
25 agitation, in the presence of a surfactant and, if necessary, another solvent.

It would therefore be desirable to have the use of homogeneous compositions which are easy to produce, do not require thermal energy in the form of heating and can be readily recycled.

Micro-emulsions are homogeneous compositions but they contain
30 significant concentrations of formulating agents, in particular emulsifiers and hydrotropic agents.

It would also seem desirable to be able to produce these stable micro-emulsions with the minimum of components other than water and the active agent to be emulsified.

WO-A-91/00893 describes paint and varnish strippers, optionally
 5 containing water, which are produced in the form of gels or bases with which organic clays such as bentones are impregnated. These compositions require the use of at least 10% of another water-soluble solvent of the N-methyl pyrrolidone type.

After lengthy research, the applicant has discovered, surprisingly,
 10 that new compositions, in the form of solutions or dispersions of a concentrated micro-emulsion type or a hydrophobic active agent or an N-alkyl pyrrolidone with water, satisfying the above criteria were able to be produced easily, using nothing more than a glycol ether or preferably a mixture of two glycol ethers.

Accordingly, the objective of the present application is to propose a
 15 composition in the form of a liquid/liquid dispersion of the micro-emulsion type or a solution of an active agent which is concentrated and dilutable in water, characterised in that it is transparent like water and essentially consists of

- at least 10 % by weight of a hydrophobic active agent having a KB (Kauri butanol) index higher than or equal to 30 and preferably 40 or less than
 20 10% of an N-alkyl pyrrolidone and
- a glycol ether or preferably a mixture of two glycol ethers, the first glycol ether in the latter case having a HLB 0.8, preferably 0.9 and more particularly 1.0 higher than that of the second glycol ether.

In the present application and the description below, the term
 25 « essentially » is used to mean at least 90 % by weight of the composition, preferably at least 95 % and more particularly at least 98 %.

By « hydrophobic » is meant that the active agent has a low solubility in water. The water-solubility of the main active agent or agents will preferably be less than or equal to 8%, in particular less than or equal to 6%,
 30 more particularly less than or equal to 4%, most especially less than or equal to 1%.

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If the composition is a micro-emulsion, it is for example water in oil (W/O), in particular « multiple » (O/W/O or W/O/W), and in particular oil in water (O/W).

The terms micro-emulsion used hereafter are therefore intended
5 to mean solutions unless it is obvious from the context that only micro-emulsions could be meant.

If using a single glycol ether, it will have a high HLB. Dipropylene glycol methyl ether is more particularly used.

If two glycol ethers are used, dipropylene glycol methyl ether is
10 used more specifically as the first glycol ether.

In order of decreasing HLB value as calculated by the Davies method, dipropylene glycol methyl ether (8.2), propylene glycol n-propyl ether (7.3), dipropylene glycol n-propyl ether (7.2), propylene glycol n-butyl ether (6.9) can for example be described.

The glycol ethers could for example represent 50 % to 90 % by
15 weight of the concentrated micro-emulsion for example, in particular 60 % to 90 %, and more especially 70 % to 90 %.

If two glycol ethers are used, the ratio of the first glycol ether to the second glycol ether may range for example from 1:8 to 4:5, in particular from
20 1:5 to 3:5, and more especially from 1:3 to 2:5 by weight.

The active agent with a KB index which is preferably greater than or equal to 30 and in particular 40 will be chosen from among fatty substances, terpenic derivatives of synthetic, semi-synthetic or natural origin, rectified or not (by distillation) such as essential oils of pine, terpenes of citrus fruits, in
25 particular orange such as d-limonene, non-water-soluble organic solvents of aliphatic petroleum origin such as the isoparaffins, certain carboxylated derivatives such as esters such as dimethyl esters, certain amides, carbonyl derivatives such as certain ketones, certain aldehydes, polar solvents other than terpenic alcohols, fatty alcohols (thus those having a high molecular
30 weight), thiols, certain amines and ethers having for example 4 to 15 carbon atoms, preferably 5 to 14 carbon atoms, in particular 7 to 13 carbon atoms.

The active agent will preferably be selected from among the citrus terpenes, in particular orange; d-limonene is more especially preferred.

The active agent will more especially be d-limonene, an RPDE mixture of dimethyl esters or a mixture thereof.

5 They can also be active agents used in cosmetics, such as a hydrating agent like Evening Primrose oil, a soothing and scar-healing agent such as allantoin, an anti-UV agent such as phenoxyethanol or mexoryl®, an anti-ageing agent such as retinol, retinaldehyde, vitamin A acid, an exfoliating agent, an antioxidant or an active pharmaceutical agent which may be used in
10 dermatology such as a corticosteroid.

The N-alkyl pyrrolidone is preferably N-methyl pyrrolidone.

It is obvious to a person skilled in the art that whenever this application talks of « a » given product, this is intended to mean « at least one » given product where justified by the context. This applies, for example, to the
15 active agent, which may be a mixture of active agents, or to the emulsifiers.

The above micro-emulsions or solutions may also contain one or more conventional additives chosen for example from additives which are soluble in a non-aqueous phase such as perfumes and preservatives such as formol or the parabens. These can be used in the standard proportions generally used in
20 micro- emulsions, in particular less than 5 %, especially 0.1 % to 3 %, and quite especially 0.1 % to 2 % by weight.

The additive might also be a preparation based on dimethylamides of unsaturated fatty acids such as that sold by BUCKMAN under the name of DMAD or BUSPERSE® 47.

25 The additives incorporated in the micro-emulsions or solutions described above impart specific properties to them such as biocidal properties. They may be rendered totally water-dilutable by adding surfactants of an emulsifying nature such as those sold by RHODIA under the name of Rhodoclean HP and/or ASP (alkoxylated fatty alcohols, derivatives of pine
30 terpenes) and Rhodasurf T-50 (ethoxylated fatty alcohol) or by using an ether amine oxide.

The present application also relates to a concentrated micro-emulsion or solution as outlined above, characterised in that it is diluted so that the diluting phase, such as an isoparaffin solvent and in particular an aqueous phase, represents up to 99 % by weight of the diluted micro-emulsion or solution, in particular up to 80 %, preferably up to 60 %, more particularly up to 50 %, most particularly up to 40 %.

The present application also relates to a method of preparing a concentrated micro-emulsion or solution as outlined above, characterised in that the liquid active agent or active agents are mixed with the glycol ether or ethers under agitation in order to emulsify or dissolve them in the latter.

Although this preparation can be carried out hot, it has a remarkable advantage of being able to be carried out at ambient temperature and quickly, within a few minutes stirring, for example using a stirrer with 1 or 2 blades, that is to say the process requires a minimum of energy.

However, a non-liquid active agent should be melted or solubilized beforehand to convert it to liquid form.

The present application also relates to a method of preparing a diluted micro-emulsion, characterised in that the diluent and glycol ether or ethers are mixed with the liquid active agent or active agents to be emulsified under agitation. The mixing order is not critical. Under preferred conditions, a preliminary mixture of the diluent and glycol ether or ethers is prepared under agitation and the liquid active agent or active agents to be emulsified are then added progressively under continued agitation. By adding « progressively » is meant, for example, adding 10% V/V per minute.

The diluent is water or an aqueous phase in particular. It may also be an isoparaffin solvent in particular. It may also be a non-water-soluble aromatic organic solvent such as solvents of petroleum origin of the type such as white spirits.

A diluted micro-emulsion or solution may also be prepared by diluting a concentrated micro-emulsion or solution as outlined above.

This being the case, the glycol ether or ethers represent 15 % to 85% by weight of the diluted micro-emulsion, for example, in particular 20 % to 80 %, more particularly 25 % to 70 % and most especially 30 % to 50 %.

The aqueous phase, if used, is preferably constituted by water, in particular demineralised water. If it is not demineralised, the aqueous phase will preferably also contain a softening agent such as a phosphonate or the tetrasodium salt of ethylene diamine tetracetic acid. For a tap water of average hardness, for example, the softening agent may represent in the order of 0.5% by weight of the composition.

If desired, the aqueous phase may also contain one or more water-soluble compounds such as colorants.

To prepare standard micro-emulsions of solvents in water, it is necessary to adjust each of the constituents depending on the concentration of the solvents. It should be pointed out that one of the other remarkable qualities of the compositions according to the invention is that if two glycol ethers are used, the content of the second glycol ether merely has to be adjusted to enable the content of active agent(s) to be varied. Accordingly, it is possible to make micro-emulsions with different contents of active agent(s) without having to devise a specific formulation every time, i.e. without having to vary several parameters on the basis of the nature and quantity of the substances contained in the formulation.

The compositions proposed by the invention also exhibit a high degree of rinsability in water, in particular the active agents which do not exhibit this property by nature, and/or a capacity for aqueous dilution and phase separation in a manner that can be controlled and predetermined as desired.

The micro-emulsions or solutions proposed by the invention readily lend themselves to recycling. After use, the volume of non-aqueous effluents to be treated can be reduced by a simple aqueous dilution. The spent solution or micro-emulsion rapidly separates into two totally distinct phases, one of which is non-aqueous. As the phases separate, the glycol ethers are distributed within

each phase to facilitate the transfer and solubilization of the respective impurities and assist in producing a clearly defined interface.

The system proposed by the invention is totally reversible. The non-aqueous phase may be filtered and/or rectified with a view to recycling. Simply
5 adding propylene glycol methyl ether and water will enable the initial product to be recreated.

This recycling process can be reinitiated at several points.

Using glycol ether(s) as proposed by the invention enables micro-emulsions of active agents to be obtained which are as different in chemical
10 nature and polarity as d-limonene and mixtures of dimethyl esters, even if the same composition is used for the rest of the formula.

These properties are illustrated in the experimental part below.

The micro-emulsions proposed by the invention enable an excellent surface preparation to be made for mechanical parts prior to assembly or can be
15 applied as a protective coating. They may be used on most metals, alloys, plastics materials and elastomers, particularly for the purpose of external maintenance on aircraft.

They enable compositions to be prepared which are excellent in terms of chemical innocuousness for use on joints and sensitive materials
20 (polycarbonates, EPDM). After use, a surface will be non-greasy with no impurity or residual film.

It is also possible to regulate the rate at which the solvents used as active agents evaporate and they generally conserve a flash point higher than
61° C; this increases with the content of diluent phase, particularly water.
25 Accordingly, the performance obtained is equal to that obtained with the halogenated solvents known from the prior art but without their harmful effects on the environment.

These properties warrant the use of the micro-emulsions or solutions proposed by the invention as an industrial solvent, a cleaner-renovator
30 or as a surface treatment for plastic materials such as garden furniture, a cleaner-renovator for stained or darkened wood and as a cleaner for polyurethane foams.

They also justify use of the micro-emulsions or solutions proposed by the invention as a means of removing residues of mastic, cleaning paint brushes, removing glues and adhesives and diluting all types of paint. They also find applications in the nautical field (washing woods, plastic materials, mechanical elements, ...) especially as they can be rinsed with seawater unlike detergents, or alternatively for stripping in the case of those containing a N-alkyl pyrrolidone. They can likewise be used to maintain and renovate painted facades, roofing, boarding, aluminium and wooden joinery, for removing certain types of graffiti from buildings, cleaning pedestrian walkways, market places, public places, lorries and household refuse containers, fixtures and furnishings on the public highways, washing the exterior of vehicles and boats, cleaning textiles such as dusters, work clothes, in particular operative sites, blinds, floor coverings, carpets and rugs, removing the varnish from and defluxing electronic printed circuit boards. They also find applications in printing, in particular for cleaning screens, particularly made from silk.

The invention therefore also relates to washing or stripping agents containing a diluted micro-emulsion as proposed by the invention or a concentrated micro-emulsion or solution as defined above.

The preferred conditions of use described in relation to the solutions, micro-emulsions and concentrates described above also apply to the other aspects of the invention mentioned above.

The examples set out below illustrate the present application.

EXAMPLES 1 to 3

Diluted micro-emulsions were prepared using the following formula:

| | Content by % Weight/ Weight | | |
|-----------------------------------|-----------------------------|---------------------|---------------------|
| | Ex. 1 | Ex. 2 | Ex 3 |
| Demineralised water | to make up 100 % | to make up 100 % | to make up 100 % |
| Dipropylene glycol methyl ether | 25.0 % | 80.0 % | 85.0 % |
| Propylene glycol n-butyl ether | 0 | 0 | 0 |
| D-limonene | 0 | 2 to 10 % | 10 % |
| RPDE mixture of dimethyl esters * | 2 to 10 % | 0 | 0 |

* Mixture of dimethyl esters sold by the RHODIA Company under the name of RPDE (dimethyl glutarate, succinate and adipate).

5 The micro-emulsions listed above were prepared as follows:

Whilst applying constant agitation, dipropylene glycol methyl ether is incorporated very progressively in demineralised water over a period of 5 minutes until the mixture is transparent. Still under constant agitation, the RPDE mixture of dimethyl esters or d-limonene is incorporated. Agitation is continued for a further 5 minutes, checking to ensure that the micro-emulsion is homogeneous, by observing its transparency for example.

It may be observed that using 25 % by weight of the single dipropylene glycol methyl ether will enable from 2 to 10 % of the RPDE dimethyl ester mixture to be emulsified. Using this single ether in a proportion of 80 % will emulsify from 2 to 10 % of d-limonene. The amount of water is the quantity needed to make up 100 % by weight in both cases.

EXAMPLES 4 to 6

Diluted micro-emulsions were prepared using the following formula:

| | Content by % Weight/ Weight | | |
|---------------------------------|-----------------------------|---------------------|---------------------|
| | Ex. 4 | Ex. 5 | Ex 6 |
| Demineralised water | to make up 100 % | to make up 100 % | to make up 100 % |
| Dipropylene glycol methyl ether | 27.0 % | 26.0 % | 45.0 % |
| Propylene glycol n-butyl ether | 10.0 % | 5 to 15 % | 25.0 % |
| D-limonene | 0 | 0 | 10 % |
| Mixture of dimethyl esters RPDE | 2 to 20 % | 15.0 % | 0 |

These micro-emulsions were prepared as follows:

Whilst applying constant agitation, the dipropylene glycol methyl ether and the propylene glycol n-butyl ether are incorporated very progressively in demineralised water for a period of 5 minutes. Still applying constant agitation, the RPDE mixture of dimethyl esters or d-limonene is incorporated. Agitation is continued for a further 5 minutes and a check is made to ensure that the micro-emulsion is homogeneous, by observing its transparency for example.

It may be observed that a composition containing 27 % dipropylene glycol methyl ether and 10 % of propylene glycol n-butyl ether will enable from 2 % to 20 % of the RPDE dimethyl ester mixture to be emulsified without having to make any adjustment to the formula other than the amount of water needed to make up a quantity of 100 % by weight.

It may also be observed that a stable micro-emulsion is obtained, containing 15 % of RPDE dimethyl ester mixture using a mixture of 26 % of dipropylene glycol methyl ether with from 5 to 15 % of propylene glycol n-butyl ether. Here too, water is added to make up the quantity to 100 % by weight.

It may also be noted (see examples 3 and 6) that a stable micro-emulsion as proposed by the invention can be prepared from 10 % of d-limonene containing 5 to 20 % of water if using 85 % of dipropylene glycol methyl ether in the first instance and 45 % of this glycol plus 25 % of propylene glycol n-butyl ether in the second instance.

EXAMPLES 7 and 8

Concentrates were prepared for a micro-emulsion using the following formula:

5

| | Content by % Weight/ Weight | |
|---------------------------------|-----------------------------|--------|
| | Ex. 7 | Ex. 8 |
| Dipropylene glycol methyl ether | 48.0 % | 62.0 % |
| Propylene glycol n-butyl ether | 22.0 % | 20.0 % |
| RPDE mixture of dimethyl esters | 30.0 % | 18.0 % |

These micro-emulsions were prepared by mixing the dipropylene glycol methyl ether and propylene glycol n-butyl ether for 5 minutes until the mixture is clear, followed by the RPDE mixture of dimethyl esters whilst continuing the constant agitation. Agitation is continued for a further 5 minutes and a check is made to ensure that the micro-emulsion is homogeneous.

After Example 7 is diluted 1/1 with water, the diluted micro-emulsion of Example 5 can be obtained if desired, containing 11.0 % of propylene glycol n-butyl ether.

The micro-emulsion of Example 8 can be diluted in any proportion, particularly in water.

EXAMPLES 9 to 12

Diluted micro-emulsions were prepared as specified in examples 1 to 3 in compliance with the following formula:

| | Content by % Weight/ Weight | | | |
|---------------------------------|-----------------------------|--------|--------|--------|
| | Ex. 9 | Ex. 10 | Ex. 11 | Ex. 12 |
| Demineralised water | 53.0 % | 10.0 % | 49.0 % | 20.0 % |
| Dipropylene glycol methyl ether | 27.0 % | 80.0 % | 26.0 % | 45.0 % |
| Propylene glycol n-butyl ether | 10.0 % | - | 10.0 % | 25.0 % |
| D-limonene | - | 10.0 % | - | 10.0 % |
| RPDE mixture of dimethyl esters | 10.0 % | - | 15.0 % | - |
| | | | | |
| Water | 34.0 % | 6.0 % | 12.0 % | 10.0 % |

The last row of the table represents the quantity of aqueous phase, for example water, from which the organic phase is made to salt out and the micro-emulsions assume a capacity to absorb water for example, i.e. can be diluted.

EXAMPLE 13: Salting out test

Having then used, for example, 100 g of the original micro-emulsion obtained in Example 6 (representing 100% by weight) to degrease mechanical car components, 40 ml of water were added to the original soiled micro-emulsion, which was shaken and then left to settle, as a result of which a cloudy appearance was observed, which was evidence that the composition was losing its homogeneity, after which a splitting of phases was observed whereby two clear phases were formed one on top of the other with a very marked separation on a level with the interface. The hydrophilic phase represents 92 ml for 65.3 ml at the outset. Since 40 ml of water were added, approximately 80% of the initial hydrophilic phase was therefore recovered.

Due to the properties of the compositions proposed by the invention, only a reduced volume of liquid has to be treated.

Furthermore, a very large proportion of organic top phase (propylene glycol n-butyl ether and d-limonene) can also be recovered and used to prepare a fresh initial micro-emulsion.

From an initial micro-emulsion comprising water (40 %), dipropylene glycol methyl ether (24 %), propylene glycol n-butyl ether (15 %), RPDE mixture of dimethyl esters (21 %), approximately 95.5 % of the initial hydrophilic phase was recovered by adding 40 ml of water.

5

EXAMPLES 14 to 18

Diluted micro-emulsions were prepared as specified in examples 1 to 3 based on the following formula:

| | Content by % Weight/ Weight | | | | |
|-----------------------------------|-----------------------------|--------|--------|--------|--------|
| | Ex. 14 | Ex. 15 | Ex. 16 | Ex. 17 | Ex. 18 |
| Demineralised water | 20.0 % | 25.0 % | 62.5 % | 20 % | 67.5 % |
| Dipropylene glycol methyl ether | 45.0 % | 37.5 % | 20.0 % | 40.0 % | 15.0 % |
| Propylene glycol n-propyl ether | 25.0 % | 27.5 % | - | - | 7.5 % |
| Dipropylene glycol n-propyl ether | - | - | 7.5 % | 30 % | - |
| D-limonene | * | 10.0 % | - | 10.0 % | - |
| RPDE mixture of dimethyl esters | * | - | 10.0 % | - | 10.0 % |

10

* Ex. 14: 10 % of RPDE mixture of dimethyl esters or d-limonene

This Example 14 demonstrates that by using glycol ether(s) as proposed by the invention, a micro-emulsion of active agents can be obtained which are as varied in terms of chemical nature and polarity as d-limonene and the dimethyl ester mixtures.

15

EXAMPLE 19

A micro-emulsion was prepared in accordance with the formula given for Example 18 but the RPDE mixture of dimethyl esters was replaced by a 5% mixture of said RPDE mixture of dimethyl esters and 5 % of d-limonene.

20

CLAIMS

1. A composition in the form of a liquid/liquid dispersion of the micro-emulsion type or solution of an active agent, concentrated and dilutable,
5 characterised in that it is transparent like water and essentially consists of
- at least 10 % by weight of a hydrophobic active agent having a KB (Kauri butanol) index greater than or equal to 30 or less than 10 % of a N-alkyl pyrrolidone and
 - a glycol ether or a mixture of two glycol ethers, the first glycol ether in the
10 latter case having a HLB 0.8 greater than that of the second glycol ether.
2. A concentrated composition as defined in claim 1, characterised in that the hydrophobic active agent is a hydrophobic active agent with a KB index greater than or equal to 40.
3. A concentrated composition as defined in claim 1 or 2,
15 characterised in that it contains a mixture of two glycol ethers.
4. A concentrated composition as defined in one of claims 1 to 3, characterised in that it is in the form of a micro-emulsion.
5. A concentrated composition as defined in one of claims 1 to 4, characterised in that it contains dipropylene glycol methyl ether as a glycol ether.
- 20 6. A concentrated composition as defined in claim 5, characterised in that it contains propylene n-propyl ether, dipropylene glycol n-propyl ether or propylene glycol n-butyl ether as a second glycol ether.
7. A composition as defined in one of claims 1 to 6, characterised in that it contains 50 % to 90 % by weight of glycol ether or ethers.
- 25 8. A composition as defined in one of claims 1 to 6, characterised in that it is diluted so that the diluent phase represents up to 99 % by weight of the micro-emulsion or diluted solution.
9. A method of preparing a concentrated composition as defined in one of claims 1 to 7, characterised in that the liquid active agent or active
30 agents are mixed under agitation with the glycol ether or ethers in order to emulsify or solubilize the latter.

10. A washing or stripping agent comprising a concentrated or diluted composition as defined in one of claims 1 to 8.

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TITLE OF THE INVENTION

Concentrated and dilutable solutions or dispersions, method of preparing same and applications thereof

ABSTRACT

A composition in the form of a micro-emulsion or solution of an active agent, concentrated and dilutable, characterised in that it is transparent like water and essentially consists of

- at least 10 % of a hydrophobic active agent having a KB (Kauri butanol) index greater than or equal to 30 or a N-alkyl pyrrolidone and
- a glycol ether or a mixture of two glycol ethers, the first glycol ether in the latter instance having a HLB 0.8 higher than that of the second glycol ether.

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Combined Declaration for Patent Application and Power of Attorney

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name; and that I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

CONCENTRATED OR DILUTABLE SOLUTIONS OR DISPERSIONS. PREPARATION METHOD AND USES

the specification of which (check one)

- [] is attached hereto;
 [] was filed in the United States under 35 U.S.C. §111 on , as
 U.S. Appln. No. _____*; or
 [X] will filed in the U.S. under 35 U.S.C. §371 by entry into the U.S. national stage of an international (PCT)
 application, PCT/FR00/00118; filed 20 January 2000, entry requested on July 23, 2001*; national stage
 application received U.S. Appln. No. _____*; §371/§102(e) date _____* (* if
 known)

and was amended on July 23, 2001 (if applicable).

(include dates of amendments under PCT Art. 19 and 34 if PCT)

I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above; and I acknowledge the duty to disclose to the Patent and Trademark Office (PTO) all information known by me to be material to patentability as defined in 37 C.F.R. §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §§ 119 (a)-(d) and 365 (b) of any prior foreign application(s) for patent, inventor's or plant breeder's rights certificate(s), or under §365(a) of any PCT application which designated at least one country other than the U.S., listed below:

| Application No. | Country | Filing Date (MM/DD/YYYY) |
|-----------------|---------|--------------------------|
| 99/00793 | France | January 21, 1999 |

If I claimed foreign priority above, I hereby identify below any foreign application for patent (including an international (PCT) application designating a country other than the United States) or for an inventor's or plant breeder's certificate, having a filing date before that of the earliest application from which foreign priority is claimed (if left blank, then there are none):

| Non-Priority Application No. | Country | Filing Date (MM/DD/YYYY) |
|------------------------------|---------|--------------------------|
| | | |

I hereby claim the benefit under 35 U.S.C. §119(e) of any United States provisional applications listed below:

| Application No. | Filing Date (MM/DD/YYYY) |
|-----------------|--------------------------|
| | |

I hereby claim the benefit under 35 U.S.C. §120 of any prior U.S. non-provisional application(s) or under §365(c) of any prior PCT international application(s) designating the U.S., listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in such U.S. or PCT international application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose to the PTO all information which is material to patentability as defined in 37 C.F.R. §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

| Application No. | Filing Date (MM/DD/YYYY) | Status (patented, pending, abandoned) |
|-----------------|--------------------------|---------------------------------------|
| | | |

As a named inventor, I hereby appoint the following registered practitioners to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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Page 2 of 2 Pages

Atty. Docket: ANGELI=2

Title: CONCENTRATED OR DILUTABLE SOLUTIONS OR DISPERSIONS. PREPARATION METHOD AND USESU.S. Application filed July 23, 2001 Serial No. _____
PCT Application filed January 20, 2000 Serial No. PCT/FR00/00118

The undersigned hereby authorizes the U.S. Attorneys or Agents appointed herein to accept and follow instructions from RINUY, SANTARELLI as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. Attorneys or Agents and the undersigned. In the event of a change of the persons from whom instructions may be taken, the U.S. Attorneys or Agents appointed herein will be so notified by the undersigned.

I hereby further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

| | | | |
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| RESIDENCE | | CITIZENSHIP | |
| POST OFFICE ADDRESS | | | |
| FULL NAME OF SIXTH JOINT INVENTOR | | INVENTOR'S SIGNATURE | DATE |
| RESIDENCE | | CITIZENSHIP | |
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